भारतीय मानक
Indian Standard

IS 3229 : 2020

नेफ़थिओनिक एसिड (सोडियम साल्ट) — विशिष्टि

(तीसरा पुनरीक्षण)

Naphthionic Acid (Sodium Salt) — Specification

(Third Revision)

ICS 71.080.40

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

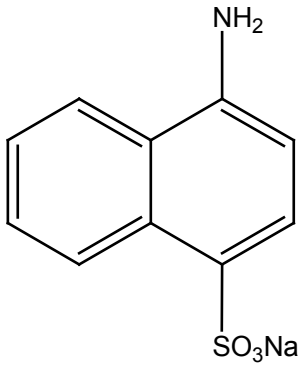
December 2020 **Price Group 4**

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was revised in 1973 and 2003. This revision has been carried out to incorporate internationally accepted analytical methods using sophisticated instruments. Purity and α-naphthylamine content is determined by High Performance Liquid Chromatography.

Naphthionic acid (sodium salt) ($C_{10}H_8O_3NSNa$), which is commonly known as sodium naphthionate and chemically named as 4-atnino-l -naphthalene sulphonic acid, monosodium salt is an important dye intermediate and is widely used in the manufacture of azo dyes. It is represented by the following structural formula:



Naphthionic Acid (Sodium Salt)

(Molecular Mass: 245.2)

(CAS Registry Number: 130-13-2)

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

NAPHTHIONIC ACID (SODIUM SALT) — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for naphthionic acid (sodium salt).

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIRMENTS

3.1 Description

The material shall be in the form of pinkish-grey to grey powder when anhydrous. The hydrated product is pinkish crystalline and contains some lumps.

3.2 The material shall also comply with the requirements of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers like jute bags or drums (*see* IS 2552) lined with polyethylene film or as agreed to between the supplier and the purchaser

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Lot or batch number;
d) Tare, gross and net mass;
e) Recognized trade-mark, if any; and
f) The minimum cautionary notice worded asunder:

'IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS'

4.2.1 *BIS Certification Marking*

Each container may also be marked with the Standard Mark

4.2.1.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau*

Table 1 Requirements for Naphthionic Acid (Sodium Salt)

(*Clauses* 3.2,5.3.1, 5.3.2 *and* 6.1)

S1 No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	Clause of IS No.
(1)	(2)	(3)	(4)	(5)
i)	Purity by NV, percentage, *Min*	75	A	
ii)	Purity by HPLC (on dry basis),percentage, *Min*	98	B	
iii)	α-naphthylamine content, by HPLC, *Max*, ppm	100	C	
iv)	Matter insoluble in water, percent by mass, *Max*	0.3	—	**11** of IS 5299

of Indian Standards Act, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 *Number of Tests*

Tests for assay, α-naphthylamine content and matter insoluble in water shall be performed on the composite sample.

5.3 *For Individual Samples*

5.3.1 The lot shall be declared as conforming to the requirements of this standard, if each of the individual test results satisfies the relevant requirements given at **3.1** and in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of matter insoluble in water and description while tested on the composite sample, the test result shall satisfy the relevant requirements given in Table 1 and **3.1.**

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE— 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis

ANNEX A

[*Table* 1, *Sl No.* (i)]

METHODS OF TEST FOR NAPHTHIONIC ACID (SODIUM SALT)

A-1 ASSAY DETERMINATION BY NITRITE VALUE

A-1.1 Reagent

A-1.1.1 *Concentred Hydrochloric Acid*

A-1.1.2 *Potassium Bromide*

A-1.1.3 *Standard Sodium Nitrite Solution* — 0.1N.

A-1.1.4 *Potassium Starch Iodide Indicate*

A-1.1.5 *Sodium Hydroxide 10 percent solution*

A-1.2 Procedure

Weigh accurately about 10 to 11 g of the sample and dissolve it in water and sodium hydroxide solution until distinctly alkaline to phenolphthalein paper. Transfer the solution to a 1 000 ml volumetric flask and dilute up to the mark with water. Mix well. Pipette 100 ml aliquot of this solution in one litre beaker add about 200 ml of water stirrer it on the magnetic add 35 ml of hydrochloric acid, 5.0 g of potassium bromide and washed ice. Cool to 10 to 15°C. Titrate, while stirring mechanically, with 0.1N sodium nitrite solution using potassium starch iodide test paper. The end point is reached when a blue-coloured ring appears which can be obtained repeatedly for a period of 10 minutes without further addition of nitrite solution. Note Burette Reading.

A-1.3 Calculation

Percent Purity (w/w) =

$$\frac{\text{Burette Reading} \times \text{Normality of } NaNO_2 \times 245.2 \times 10}{\text{Weight of Sample (in g)} \times 10}$$

ANNEX B

[*Table* 1, *S1No.* (ii)]

DETERMINATION OF PURITY OF NAPHTHIONIC ACID SODIUM SALT BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-0 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-1 APPARATUS

Binary Gradient Liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2 COLUMN

C18 100A 250 × 4.6mm 5µm or equivalent.

B-3 REAGENT

a) Acetonitrile — HPLC grade,
b) Water — HPLC Grade,
c) Tetra butyl ammonium hydrogen sulphate — HPLC Grade, and
d) Naphthonic acid sodium salt — Known purity.

B-4 STANDARD PREPARATION

Weigh accurately 0.0500 g standard naphthonic acid sodium salt in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

B-5 SAMPLE PREPARATION

Weigh accurately 0.0500 g sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

B-6 BUFFER PREPARATION

0.2 percent tetra butyl ammonium hydrogen sulphate in HPLC grade Water.

B-7 FLOW RATE

1.00 ml/min

B-8 MOBILE PHASE

Acetonitrile	Buffer
35	65

B-9 COLUMN OVEN TEMPERATURE

40°C

B-10 INJECTION VOLUME

5µl

B-11 RUN TIME

15 min

B-12 WAVE LENGTH

220 nm

B-13 PEAK TIME

Naphthonic Acid Sodium salt - 4.56 min

B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to naphthonic acid sodium salt on the chromatogram of the material. the concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with standard naphthonic acid sodium salt.

Percent of Naphthenic Acid Sodium salt

$$= \frac{A2 \times V1 \times W1 \times B2}{A1 \times V2 \times W2 \times B1} \times 100$$

where

A1 — area of STD naphthonic acid sodium salt,

V1 — injection volume of STD naphthonic acid sodium salt,

W1 — weight of STD naphthonic acid sodium salt,

B1 — total volume of STD naphthonic acid sodium salt,

A2 — area of naphthonic acid sodium salt peak in sample,

V2 — injection volume of sample,

W2 — weight of sample, and

B2 — total volume of sample.

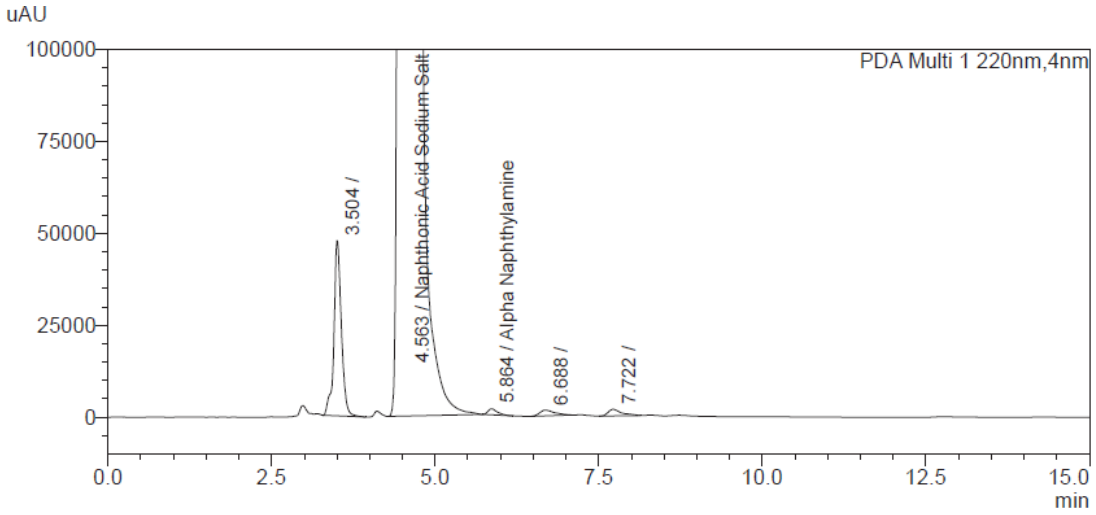


FIG. 1 TYPICAL CHROMATROGRAM

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF α-NAPHTHYLAMINE by HPLC

C-1 OBJECTIVE

To determine α-Naphthylamine in naphthonic acid sodium salt by high performance liquid chromatography.

C-2 REAGENT

a) Acetonitrile —HPLC grade,
b) Water — HPLC Grade,
c) Tetra butyl ammonium hydrogen sulphate — HPLC Grade, and
d) α-Naphthylamine — Known purity.

C-3 APPARATUS

Binary gradient liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-4 COLUMN

C18 100A 250 × 4.6 mm 5 µm or equivalent.

C-5 STANDARD PREPARATION

Weigh accurately 0.025 0 g standard β-Naphthylamine in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetoniltrile A Take 1 ml of solution A dilute 100 ml with Acetonitrile.

C-6 SAMPLE PREPARATION

Weigh accurately 0.100 0 g sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

C-7 BUFFER PREPARATION

0.2 percent tetra butyl ammonium hydrogen sulphate in HPLC grade water.

C-8 FLOW RATE

1.00 ml/min

C-9 MOBILE PHASE

Acetonitrile	Buffer
35	65

C-10 COLUMN OVEN TEMPERATURE

40°C

C-11 INJECTION VOLUME

5µl

C-12 RUN TIME

15 min

C-13 WAVE LENGTH

220 nm

C-14 PEAK TIME

Naphthonic Acid Sodium salt — 4.56 min
α-Naphthylamine — 5.86 min

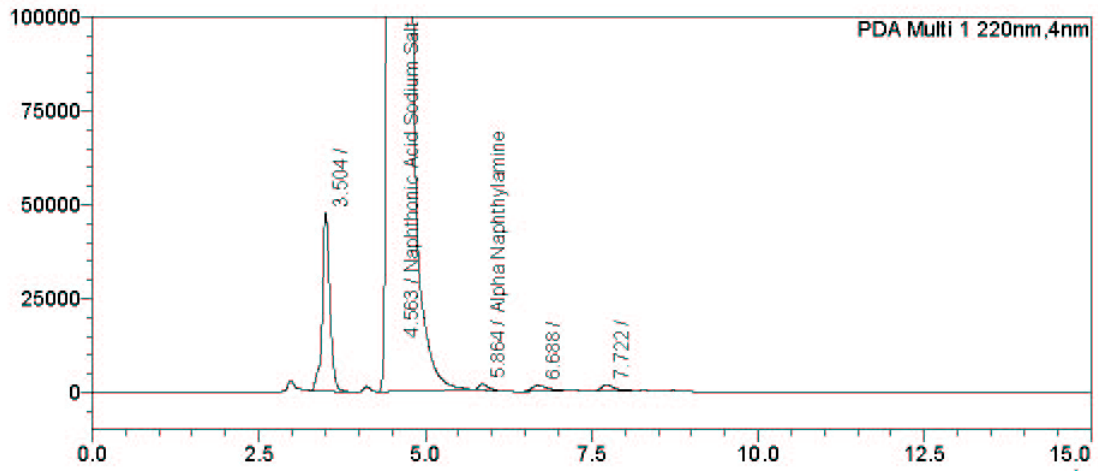


FIG. 2 TYPICAL CHROMATOGRAM

C-15 CALCULATION

Calculate the peak area of individual constituent pertaining to α-Naphthylamine on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with Standard α-Naphthylamine

Percent of α-Naphthyamine =

$$= \frac{A2 \times V1 \times W1 \times B2}{A1 \times V2 \times W2 \times B1} \times 100$$

Where

A1 = area of STD α-Naphthyamine,
V1 = injection volume of STD α-Naphthyamine,
W1 = weight of STD α-Naphthyamine,
B1 = total volume of STD α-Naphthyamine,
A2 = area of α-Naphthyaminepeak in sample,
V2 = injection volume of sample,
W2 = weight of sample, and
B2 = total volume of sample.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
ICT, Mumbai	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited - Atul – Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K. Astik(*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Smt Maitri Vyas (*Alternate*)
Department of Chemicals & Petrochemicals, Ministry of Chemicals & Fertilizers, New Delhi	Shri Jasbir Singh
Ministry Of Defence (Research and Development), New Delhi	Dr Vijay Tak
National Test House(er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr Jaychandran M. Nair
The Bombay Textile Research Association, Mumbai	Dr Ashok N. Desai

Organization	*Representative(s)*
The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar
BIS Director General	Shri N. K. Bansal, Scientist 'F' and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shri Chandrakesh Singh

Scientist 'D', BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 26 (14050).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617, 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019	265 0206, 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442, 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093	2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi